Identifier:	Revision:	Effective Date:	Review Date:
SOP-09.02	**1**	**04/11/01**	**12/16/2003**

ER Document Catalog Number: **ER2001-0127**

Author: David Vaniman

environmental restoration project

A Department of Energy Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Operating the Cameca SX-50 Microprobe

Los Alamos
NATIONAL LABORATORY
Los Alamos, New Mexico 87545

Revision Log

Revision No.	*Effective Date*	*Prepared By*	*Description of Changes*	*Affected Pages*
R0	3/16/92	Marjorie G. Snow	New procedure	all
R1	04/11/01	David Vaniman	Complete revision for operation of SX-50 microprobe	all
Review	12/16/2003	Mark Thacker	Deemed adequate.	All

Operating the Cameca SX-50 Microprobe

Table of Contents

Operating the Cameca SX-50 Microprobe

1.0 PURPOSE

This procedure provides instructions for the operation of the Cameca Instruments Incorporated, Model SX-50, electron microprobe system.

2.0 SCOPE

This SOP is a manadatory document and shall be implemented by all ER Project participants when collection electron microprobe data for the ER Project

3.0 TRAINING

3.1 All users of this SOP are trained by reading the procedure, and the training is documented in accordance with QP-2.2.

3.2 The **Geology Task Leader** will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with QP-2.2.

4.0 DEFINITIONS

4.1 *Machine Custodian* —The Machine Custodian is responsible for Cameca SX-50 maintenance and User instruction. This includes calibration of magnification marker and Energy Dispersive System (EDS) gain, EDS resolution check, software and electronically-stored system backups, and all instruction and training of Procedure Users.

4.2 *SX-50* —SX-50 is the model name of the electron microprobe manufactured by Cameca Instruments, Incorporated (purchase date 3/92, serial number 431).

4.3 *SX-50 system* — The SX-50 System includes the Cameca SX-50 electron microprobe and the Princeton-Gamma Tech (PGT) energy dispersive system.

4.4 *PGT* — Princeton Gamma-Tech (PGT) is the manufacturer of the energy dispersive system (EDS) operated as part of the SX-50 system.

4.5 *IMIX* — Integrated Microanalyzer for Imaging and X-Ray (IMIX) is the integrated software package provided by PGT to operate the energy dispersive system.

4.6 *WDS* — The SX-50 contains four Wave-length Dispersive Spectrometers (WDS).

5.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

5.1 Focus Area Leader

5.2 Team Leader

5.3 Quality Program Project Leader

5.4 Geology Task Leader

6.5 ER Project personnel

6.0 EQUIPMENT

Descriptions of equipment constituting the SX-50 system are provided below.

6.1 *SX-50* — The Cameca SX-50 (purchase date 3/92, serial number 431) is an integrated computer controlled electron microprobe with capabilities for secondary and backscattered electron imaging, wavelength- and energy-dispersive elemental acquisition, and image processing.

6.2 *PGT Energy-Dispersive System* — Princeton Gamma-Tech scintillation detector that provides energy-dispersive X-ray spectra for identification and abundance scaling of elemental constituents in samples.

7.0 PROCEDURE

Note: Subcontractors performing work under the ER Project's quality program may follow this standard operating procedure (SOP) for electron microprobe analysis or may use their own procedure(s) as long as the substitute meets the requirements prescribed by the ER Project Quality Management Plan, and have been approved by the ER Project's Quality Program Project Leader (QPPL) before the commencement of the activitie(s).

Note: ER Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

Note: Deviations from SOPs are made in accordance with QP-4.2, Standard Operating Procedure Development and documented in accordance with QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

The SX-50 system is capable of analytical work in the following general areas: elemental qualitative and semi-quantitative analysis using no standards, semi-quantitative and quantitative elemental analysis using standards, image

analysis, particle analysis, elemental mapping, elemental line profiles, and composite analysis of X-Ray maps and images. Procedure Users are responsible for determining what methods are appropriate for their work.

7.1 Sample Preparation - Samples to be examined with the SX-50 must be in a form or size that can be inserted into, or attached to, an SX-50 stage mount. Quantitative microanalyis routines assume that all samples for analysis will be relatively flat and that analysis sites will be normal to beam incidence. Before being placed in the SX-50, non-conductive samples may be given an electrically conductive coating. Carbon is most often used, however specific applications may benefit from other types of conductive coatings. Carbon coating procedures are outlined in the Ladd Vacuum Evaporator Instruction Manual.

7.2 Selection of Standards - The Procedure User may employ standards traceable to National Institute of Standards and Technology (NIST, formerly National Bureau of Standards, NBS) or well characterized materials published in credible technical journals and widely used by microanalysts for calibration of quantitative X-Ray analysis routines (e.g. Standards received from the Smithsonian, evaluated for homogeneity, and published in GeoStandards Newsletter, as in Jarosewich et.al., GeoStandards Newsletter,1980). Procedure Users may use standards from other sources for specific applications but must document the basis for usage of these standards in their notebooks. (For example, reagent-grade NaCl may be used as a Cl standard by stating the material source and noting the stoichiometric nature of this material even though it is not NIST-traceable).

7.3 Sample Insertion and SX-50 Operation - Detailed operating instructions for the SX-50 system are given in the SX-50, IMIX, and VISILOG (VISIVIEW) Manuals stored in the laboratory near the instrument. After training the Procedure User should refer to the manuals when questions arise or consult with the Machine Custodian to solve specific problems.

7.4 Data Acquisition and Reduction - Data may consist of image information and/or elemental information. Image and elemental information may be processed following instructions in the SX-50, IMIX, and VISILOG Manuals. Image and elemental information may be photographed and/or printed on paper following instructions in the SX-50 reference Manual if an electronic copy or a hard copy is desired. The Cameca software package “QuantiView” comprises software used for acquisition and reduction of quantitative elemental data. “QualiView” is the Cameca software package used for acquisition of qualitative elemental information and “VisiView” is the image processing software package. Data reduction is based on the methods of Pouchou and Pichoir (1985).

7.5 Sample Control – Sample identification will be based on the unique identifier marked on the sample. This will typically be an etched identification on the thin section that has been coated for analysis.

7.6 Potential Sources of Error and Uncertainty - Criteria for recognizing and evaluating potential sources of error and uncertainty will be indicated by the Procedure User's inability to obtain a quality image or to generate a semi-quantitative or quantitative analysis within tolerance limits. Acceptance criteria for quantitative analysis of Samples are based on acceptable analyses of appropriate standards. Procedure Users may use as a general guide a value of 2 sigma. That is, if standard analyses are within 2 sigma (based solely on counting statistics) of the list or published values, then the analysis is acceptable. Procedure Users may define different acceptance criteria (e.g. charge balance ratios).

7.7 Equipment Malfunctions - Malfunction of the SX-50 System is readily detectable by the Machine Custodian during operation of the instrument. If a trained Procedure User has doubts concerning his/her ability to detect equipment malfunction during operation of this equipment, he/she should consult with the Machine Custodian.

7.8 Safety Considerations - Normal operating conditions as performed by trained Procedure Users present no safety hazards.

7.9 Environmental Conditions - Normal interior building temperature and humidity are acceptable for the operation of the SX-50 System. Cooling water for the SX-50 diffusion pump and electronics chassis is supplied by the building chilled-water system maintained in the range of 55 to 65 degrees Fahrenheit. Ambient air temperature for the SX-50 System should range between 60 and 80 degrees Fahrenheit. If environmental conditions move out of range during operating the SX-50 system in WDS mode, Procedure Users should take extra precaution to ensure system stability by checking standards often.

7.10 Calibration of Magnification - The Machine Custodian (or delegated individual) will check the accuracy of the computer-generated micrometer marker annually against NIST Reference Material 484, SEM magnification Standard. Tolerance is + 10%. If out of tolerance it will be the Machine Custodian's responsibility to arrange for repair of the instrument so that it will be within tolerance.

7.11 Calibration of Gain - The Machine Custodian (or delegated individual) will check the gain on the PGT EDS annually by following the energy calibration instructions in the IMIX Instruction Manual using Copper $K\alpha$ and $L\alpha$ X-Ray lines. Tolerance is +20 eV. If found to be out of tolerance, it will be the Machine Custodian's responsibility to recalibrate the gain on the PGT EDS

system as described in the IMIX Manual. If the gain cannot be calibrated it will be the Machine Custodians responsibility to initiate repair for the instrument so that it will be within tolerance.

7.12 PGT Energy Resolution Check - The Machine Custodian (or delegated individual) will check the energy resolution of the PGT EDS detector annually by measuring the full width at half maximum (FWHM) of the Manganese K-alpha X-Ray line, the industry standard for detector resolution. This is a check for degradation of the detector ONLY: no adjustment or calibration will be made.

7.13 Lessons Learned

During the performance of work, ER Project personnel shall identify, document and submit lessons learned in accordance with QP-3.2, Lessons Learned. This QP can be located at: http://erinternal.lanl.gov/home_links/Library_proc.htm.

8.0 REFERENCES

ER Project personnel may locate the ER Project Quality Management Plan/ER Project QP requirements crosswalk at http://erinternal.lanl.gov/home_links/Library_proc.htm.

The following documents have been cited within this procedure:

QP-2.2, Personnel Orientation and Training

QP-3.2, Lessons Learned

QP-4.2, Standard Operating Procedure Development

QP-4.3, Records Management

QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities

Cameca SX-50 Operation Manuals: User's Guide, Reference Guide, Quantitative Reference Guide, and X-Ray Microanalysis, Geo-Multilabel, Wavelength Table Reference Manuals, 1991, Cameca, Inc. Paris, France.

IMIX Instruction Manual, 1990, Princeton Gamma-Tech, Inc., Princeton, New Jersey, USA

VISILOG User's Guide, 1988, Noesis, Jouy en Joses, France

Instruction Manual, Ladd Vacuum Evaporator, cat. no. 3000, 27 pgs., Burlington, Vermont

Jarosewich, E., J. A. Nelen, and J. A. Norberg, “Reference Samples for Electron Microprobe Analysis,” in *GeoStandards Newsletter*, Vol. 4, No. 1, p. 43, April 1980.

Pouchou, J. L. and F. Pichoir, “PAP” $\Phi(\rho Z)$ Procedure for Improved Quantitative Microanalysis,” in *Microbeam Analysis-1985*, J. T. Armstrong, Ed., San Francisco Press, Inc., San Francisco, California, USA, p.104.

9.0 RECORDS

The **Procedure User** is responsible for submitting the following records (processed in accordance with QP-4.3) to the Records Processing Facility.

9.1 Notebook records of the sample handling and results of analysis relevant to production of electron microprobe data.

9.2 Data submittals for the ER electronic database

Using a token card, click here to record "self-study" training to this procedure.

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.